Beamline U10B Startup Procedure

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1. SETTING UP THE EXPERIMENTS

1.1 Preparing for Operation

1.1.1 Enabling the Beamline

There are several things you will need before the beamline can be enabled:

1. The safety checklist must be completed and signed.

2. Everyone present at the beamline must be wearing a valid BNL ID badge.

3. Everyone present at the beamline must have competed the Beamline Operations Safety Awareness (BLOSA) training (expires every two years).

4. You should have your Safety Approval Form number available, which can be found on the web-based SAF database: http://130.199.76.84/safety/default.asp

In order to enable the beamline, page the Operations Coordinator (follow the directions by the phone) and request to have you Safety Approval Form be posted on the U10B yellow board. The Operations Coordinator will first inquire about the items listed above, and then promptly post the SAF form and enable the beamline for use.

1.1.2 Setting Up a Data Directory

1. If this is your first visit to Beamline U10B, you must first create a folder with your name within the spectra folder. A shortcut to this folder can be found on the desktop or by following:
   C Drive→ Documents and Settings→ U10B→ My Documents→ Spectra

2. Open Omnic by double-clicking the icon on the desktop.

3. Under the ATLUS menu, select “Show Atlus Window”**. If a window appears asking to initialized the microscpe stage, be sure the condenser is lowered completely before pressing F10.**

4. To setup Atlus and Omnic to automatically open to your folder when saving files, in Omnic, click on the EDIT menu and then choose OPTIONS.

5. Under the FILE tab, change the path of “Initial Spectra”, “Initial Autosave”, and “Initial Mapping” to your directory. When you are finished click OK.

6. In the FILE menu of Omnic, choose “Save Configuration As” and enter “default.con” in the Filename box. Be sure to also check “Set as default configuration.”

1.1.3 Cooling the Detector

The continuum FTIR microscope at U10B is equipped two internal detectors: MCT-A (range: 4000-650 cm^{-1}) and MCT-B (range: 4000-500 cm^{-1}, but 10 times reduced sensitivity). Choose the detector most appropriate for the experiment to be run. Liquid nitrogen can be found in the 5L dewar located at U10B. Insert the funnel into the port labeled with the selected detector (A or B). *Always used eye protection when handling liquid nitrogen.* Fill the small green thermos (~1L) with liquid nitrogen. Slowly fill the funnel using the green thermos. Fill the funnel to the top and let all the liquid nitrogen drain before filling it again. About two L will be needed to cool the detector from room temperature and fill the detector dewar. Avoid getting liquid nitrogen into the microscope by removing the funnel as soon as overflow is visible. Quickly direct the funnel to deposit the remaining liquid back into the thermos or 5L dewar. One fill should last 8-12 hours.

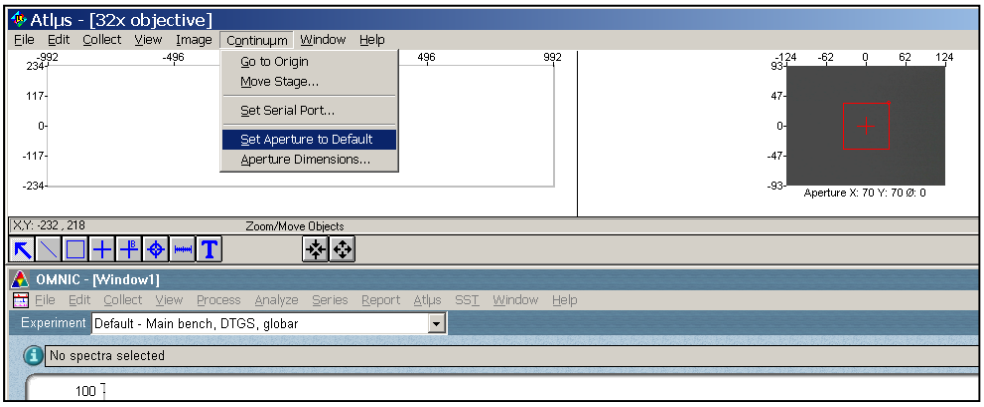
1.2 Microscope Alignment

1.2.1 Choosing Experiment Configuration

In the Omnic “Experiment” window, choose from the dropdown list the proper experiment configuration, i.e. globar or synchrotron, transmission or reflection measurement, and MCT-A or MCT-B detector.

1.2.2 Setting the Aperture:

In Atlus under the CONTINUUM menu, select “Set Aperture to Default”. The aperture should now be set at 50 x 50 μm. To set the aperture dimensions to a value other than the default, select APERTURE DIMENSIONS… in the CONTINUUM menu. After entering the x and y values you would like to use and click “Apply” and then “OK” to activate the new dimensions.

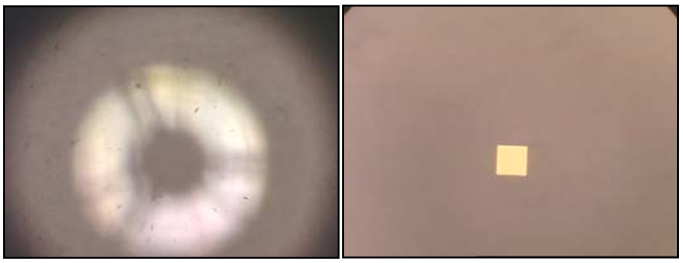


1.2.3. Aligning Microscope for Reflection Mode:

1. Select reflection mode with the blue button on the front of the microscope.
2. Insert a gold mirror onto the sample stage. Using the 10x glass objective, bring the mirror into focus. Rotate to the 32x IR objective and bring the mirror into focus again. The aperture should be centered on the crosshairs. If not, report the issue to a local contact.

1.2.4 Aligning Microscope for Transmission Mode (no sample or substrate):

1. Select transmission mode with blue button on the front of the microscope.
2. Adjust the condenser height (red-labeled knob) until aperture is in focus, when viewed from microscope eyepiece.
3. Center the aperture on the crosshairs by repositioning the condenser.

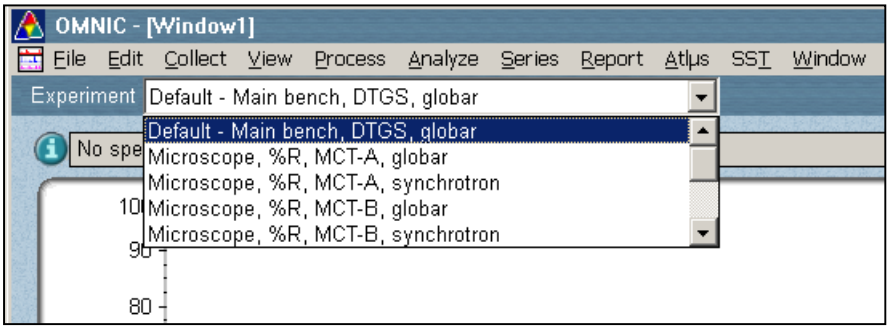


View of aperture unfocused (left) and focused (right).

1.3 Checking Signal Strength

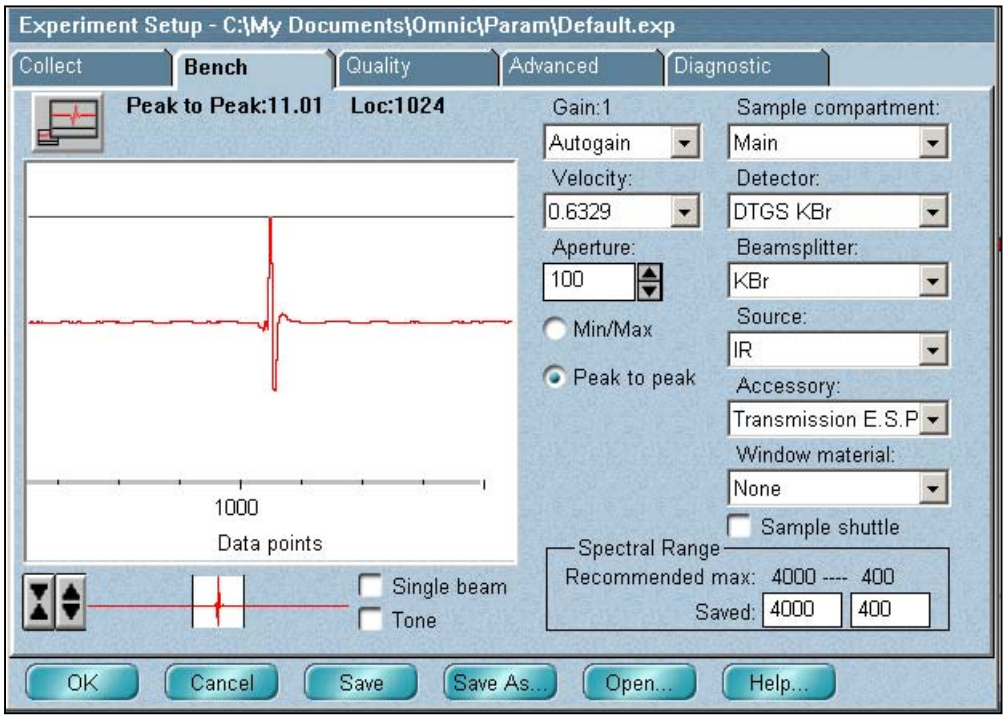
1.3.1. Check Main Bench Signal:

1. In the Omnic **"**Experiment" window, choose "Default- Main bench, DTGS, globar".



2. Under the Omnic COLLECT menu, choose EXPERIMENT SETUP.

3. Then select the BENCH tab. With the gain set at 1, the **Peak to Peak** signal of the interferogram signal should be 10-12 volts. If not, report the issue to a local contact. Click **OK**.



1.3.2 Check Signal Through Microscope with Globar:

1. In the Omnic **"**Experiment" window, choose the "Microscope/globar" experiment with the appropriate technique and detector for your experiment (example: "Microscope, %R, MCT-A, globar").

2. Under the Atlus CONTINUUM menu, select "Set Aperture Dimensions…". Set the aperture to 50 x 50 μm and click "Apply" and then "OK". The aperture should now be resized to 50 x 50 μm.

3. Under the Omnic COLLECT menu, choose EXPERIMENT SETUP.

4. Select the BENCH tab. With the gain set at 1, the peak-to-peak signal of the interferogram should fall within the range found in the table below. If not, report the issue to a local contact. Click **OK** to continue.

globar

Measurement Technique	Detector	Peak to peak range (volts)
% Reflection	MCT-A	>2.0
% Reflection	MCT -B	>0.4
% Transmission	MCT-A	>4
% Transmission	MCT-B	>1.4

1.3.3 Check Signal Through Microscope with Synchrotron:

1. In the **Omnic** "Experiment" window, choose the "Microscope/synchrotron" experiment with the appropriate technique and detector for your experiment (example: "Microscope, %R, MCT-B, synchrotron").

2. Under the Atlus CONTINUUM menu, select "Set Aperture Dimensions…". Set the aperture to 10 x 10 μm and click "Apply" and then "OK". The aperture should now be resized to 10 x 10 μm.

3. Under the Omnic COLLECT menu, choose EXPERIMENT SETUP.

4. Select the BENCH tab. With the gain set at 1, the peak-to-peak signal per amp (or at I=1000 mA) should fall within the range found in the table below (example: %T, I=546mA and p-p=9.0V, p-p/amp = 9.0V / 0.546A = 16.5). Click **OK** to continue.

synchrotron

Measurement Technique	Detector	p-p (volts) / I (amps)
% Reflection	MCT-A	>6
% Reflection	MCT-B	>.8
% Transmission	MCT-A	>10
% Transmission	MCT-B	>2.8

1.3.4 Record Signal Strength in Beamline Notebook:

1. %T: Confirm that the aperture is focused and centered. %R: Confirm that the aperture is focused onto the reflective surface.

2. Enter signal strength value in the U10B Logbook. Fill in all other information asked for in the logbook (date, detector, microscope mode, source, ring current).

1.4 Adjusting for Substrate in Transmission Mode

1.4.1 Adjusting Condenser to Correct for Substrate:

1. Note the thickness of you substrate.

2. Adjust the dial on the condenser to match the substrate thickness, in mm.

3. Refocus and realign condenser for transmission.

1.4.2 Refocusing Condenser for the Maximum Infrared Throughput

1. Under the Omnic COLLECT menu, choose EXPERIMENT SETUP and then select the BENCH tab).

2. Optimize the peak-to-peak signal by rotating the focus of the condenser slowly *clockwise*, being careful not to "crash" into the sample holder. Note- focusing the IR light may defocus the visible aperture seen from the microscope.

2. DATA COLLECTION

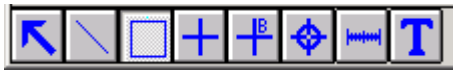
2.1 Collecting a Background

1. Identify map background point, set the aperture and optimize the signal. In the Omnic COLLECT menu under EXPERIMENT SETUP, BENCH tab, make sure the gain is set to *Autogain*.
2. Under the Atlus COLLECT menu, choose MAP SETUP.
3. Under the COLLECT tab, enter the map title, number of scans (background and sample), resolution and the final format. Then click OK.
4. Under the Atlus COLLECT menu, choose COLLECT BACKGROUND AT CURRENT LOCATION.
5. The background will then be collected.

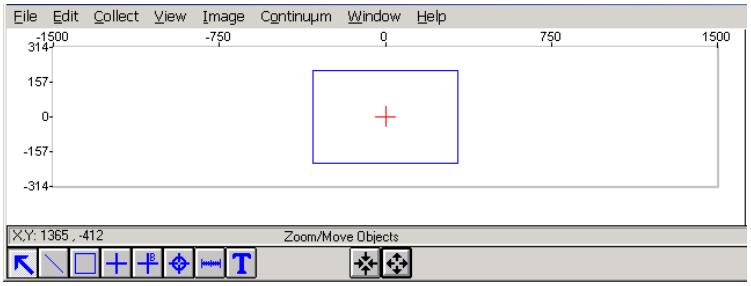
2.2 Capturing a Mosaic

1. Identify the region of the sample that you'd like to map. Center this area in the camera image window. Adjust the illumination. Turn off the aperture illumination so that a white square does not appear in all of the mosaic pixels.

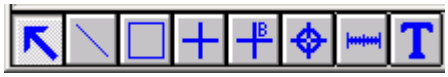
2. Using the "area map" tool (gray button at the bottom of the Atlus screen that contains a blue square), draw a square in the image area window around your sample.



3. You can click the "zoom in" or "zoom out" buttons to see that selected area in the stage area window.



4. You can use the “arrow” tool to increase the size of the mosaic area.



5. Once the area is defined, under the Atlus IMAGE menu, choose CAPTURE MOSAIC.

6. Once Atlus has captured the mosaic, save the image by clicking on SAVE MOSAIC in the Atlus IMAGE menu.

2.3 Collecting a Map

1. Using the Atlus mapping tools, located at the bottom left corner of the Atlus window, define the area you wish to map on the mosaic.

2. Save the mosaic again with the mapping area defined on it.

3. Under the Atlus COLLECT menu, choose the MAP SETUP option.

4. Click on the DIMENSIONS tab and enter the desired step size for the map. Click *Apply* and the duration of the map will appear at the bottom of the dialog box.

5. Adjust the map as needed to accommodate the time frame of the data collection.

6. Delete the values in the BACKGROUND coordinates box. If you do not do this, the microscope will collect another background spectrum when you start the map.

7. Once the correct map area is outlined click OK.

8. Confirm that the information in the COLLECT, APERTURE, and OPTIONS tabs are correct.

9. In the Omnic menu, under EXPERIMENT SETUP, BENCH tab, confirm that you have good IR signal.

10. Under the Atlus COLLECT menu, choose COLLECT MAP.

11. Once the map is completed Atlus will prompt you for a filename.

2.4 Fluorescence Imaging

** Turn on UV lamp burner and warm up for 10-15 minutes. Follow all warnings on the top of the burner unit.**

2.4.1 Sample Viewing

1. Focus on the sample with the visible light (using either the glass or IR objective).
2. Turn filter cube turret to desired filter. (If you don't know which one, try WB).
3. Open the fluorescence shutter. Blue or green light (depending on the filter) should be visible from the objective.
4. Turn off the visible light. You should be able to see your sample fluorescence.
5. For brighter fluorescence, adjust eyepiece light path completely to the eye position.
6. For the brightest fluorescence, unlock the setscrew for the dichroic mirror with a 3/32" ball driver (1/4 turn only!) and slide the dichroic mirror out.
7. To improve the brightness of the camera image, adjust the gain on the camera control box. Your options are high or low.

2.4.2 For Capturing a Mosaic

1. Capture mosaic following directions in section 2.2.

2.4.3 For Collecting IR Data

1. Dichroic mirror MUST be pushed back in and locked in place.
2. IR objective must be used.

2.5 If the Map Crashes before Finishing!

If the map crashes for some reason, Atlus automatically saves it as "default.map". In order to retrieve what has been collected, you must search for this file, copy it to your directory, and then rename it BEFORE you start collecting another map. As soon as a new map is started, it will be given the "default.map" filename and your previous map will be permanently deleted. It is not possible to resume and finish a map that crashed before its completion.

2.6 Resetting the Instrument if Microscope or Computer Stop Communicating

1. In Omnic, in the COLLECT menu, choose EXPERIMENT SETUP and the DIAGNOSTICS tab.

2. Click the RESET BENCH tab. Wait for a minute or two to see if the interferogram reappears. If not, follow the following steps.

3. Turn off IR spectrometer. (Power switch is on the back, lower left side, just below the power cord.)

4. Turn off the microscope. (Power switch is on the left hand side, towards the back, below the power cord.)

5. Shut down the computer.

6. Turn on IR spectrometer. Wait until the green “Scan” light starts blinking.

7. Turn on microscope.

8. Turn on computer. Start up Omnic and Atlus. When Atlus starts, it will ask to initialize the microscope stage. Be sure the condenser is lowered completely before pressing F10.

9. In the Omnic “Experiment” window, choose “Default- Main bench, globar, DTGS.”

10. In the Omnic COLLECT menu, choose EXPERIMENT SETUP, choose the BENCH tab, and you should see a signal again. If not contact beamline staff.

2.7 Backing up Data onto a CD at U10B

1. Insert a blank CD-RW or CD-W into the CD drive.

2. Open the program Roxio Easy CD Creator.

3. When the “Select a Project” Box appears, put the pointer over the “Make a Data CD” selection and then choose “Data CD Project”. The Roxio program will then start and the Untitled Data CD Project box will appear.

4. In the SELECT SOURCE FILE pull down menu, select the appropriate folder (your folder in the Spectra folder). All of the sub-folders should then appear in the box located under this pull down menu.

5. Click and drag the appropriate folder into the box located on the lower right hand side of the screen.

6. Repeat this step until all of your data files appear within this box.

7. Press the red RECORD button located above this box. The RECORD CD SETUP dialogue box will then appear.

8. Click on START RECORDING.

9. The CD will now burn, try not to run any other applications while the CD is burning.

10. Once the CD is done, close Roxio Easy CD Creator and remove your CD from the drive.

3. END OF BEAMTIME CHECKLIST

3.1 Leaving the Beamline Unattended (Pink Cards)

You are allowed to leave the beamline unattended while it is mapping for up to 24 hours. You must fill out a pink card (located near the U10B phone) and post it in the appropriate holder. The pink card must be completely filled out with your name, the beamline number, location and phone number where you can be reached (you may leave more than one), and the date and time you left and when you plan on returning.

3.2 End of Day/Beamtime Checklist

At the end of your beamtime you must follow the end of day checklist as follows:

1. Turn off the three illuminators on the microscope.

2. In Omnic, set experiment mode to **Default-Main Bench, Globar, DTGS**

3. Turn the TV to channel 8

4. Return tools and clean up any items that you have accumulated at the beamline

5. Close photon mask

6. Close beamline isolation valve, V1

7. Call operations coordinator and have SAF removed and beamline disabled (or post a pink card if you will be using the beamline the next day)

8. Turn off gain on camera (if used).